



## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Zhihong Jin et al.

Art Unit: 1745

Serial No. : 09/787,858

Examiner: Tracy Mae Dove

Filed : March 22, 2001

For : Performance Enhancing Additives for Electrochemical Cells

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## DECLARATION UNDER 37 CFR §1.131

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I, Michael F. Mansuetto, do hereby declare and say:

My home address is 576 Bradley Road, Bay Village, Ohio 44140.

I have a PhD degree in Inorganic Chemistry from Northwestern University, a MS degree in Inorganic Chemistry from Northwestern University, and a BS degree in Chemistry from Wheeling Jesuit College.

I have worked in the field of chemistry for 8 years.

I was employed as a chemist by Argonne National Laboratory for 2 years.

I have been employed by Eveready Battery Company, Inc., for 6 years. During that entire time I have been a Staff Technology Engineer.

I am an Applicant in the above-identified patent application.

I declare that I directed experiments in which niobium-doped  $\text{TiO}_2$  (NTO) having a resistivity less than 100 ohm-cm was produced prior to June 17, 2000. The initial criteria for the experiment included maximizing the conductivity (minimizing the resistivity) of the NTO.

Prior to November 5, 1999, I directed that an experiment be conducted in which a prior process was modified by heating the niobium-doped  $\text{TiO}_2$  in a reducing atmosphere to achieve a low resistivity. In this experiment, niobium-doped  $\text{TiO}_2$  was heated in a reducing atmosphere in an added step. A mixture of about 250 pounds of  $\text{TiO}_2$  and  $\text{Nb}_2\text{O}_5$  (85:15 ratio by weight) with PVA was wet milled, then spray dried. The dry powder was heated in air for 10 hours at  $1290^\circ\text{C}$ , followed by heating in air for 10 hours at  $1410^\circ\text{C}$ . Each of three batches of about 50 pounds of the resultant powder was then heated for 5 hours at  $1100^\circ\text{C}$  in a reducing furnace (hydrogen and nitrogen atmosphere). Testing of a sample showed the resultant NTO had a resistivity of 15 ohm-cm.

In additional experiments prior to June 17, 2000, niobium-doped  $\text{TiO}_2$  was also made by wet milling about 800 pounds of  $\text{TiO}_2$ ,  $\text{Nb}_2\text{O}_5$  and PVA, followed by spray drying. A first batch

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of about 500 pounds of the spray dried material was heated in air only once, for 10 hours at 1410°C. The powder was then heated for 5 hours at 1100°C in a hydrogen and nitrogen atmosphere. The resultant NTO had a resistivity test result of 5 ohm-cm. A second batch of about 300 pounds of the spray dried material was heated for 5 hours at 1100°C in a hydrogen and nitrogen atmosphere, without first heating in air. The NTO from the second batch had a resistivity of 72 ohm-cm.

I further declare that I obtained  $\text{Fe}_2\text{O}_3\text{-TiO}_2$  powder ( $\text{Fe}_2\text{TiO}_5$ , Stock No. 13138, CAS No. 12789-64-9) from Alfa Aesar, Ward Hill, MA, USA. When a sample of the powder was tested for resistivity, the result was about 124,000 ohm-cm.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patents issuing thereon.

Signed:

Sept. 18, 2003  
Date

Michael F. Mansuetto  
Michael F. Mansuetto